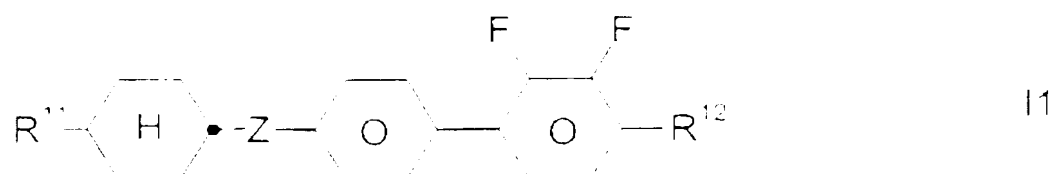


Liquid-crystalline medium

The invention is related to a liquid crystalline medium based on a mixture of compounds having positive mesogenic dielectric anisotropy, which is regulated at least one ring and at least formula II



and at least one ring and at least formula III



in which

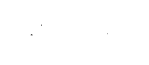
R¹¹, R¹² and R²¹ are each, independently of one another, an alkyl or alkenyl radical having up to 12 carbon atoms which is unsubstituted, substituted by one or two fluorine atoms, or at least one substituent by halogen, where one or more CH₃ groups in these radicals may also, in each case independently of one another, be replaced by a group defined by



where R¹ and R² are each, independently of one another, an alkyl or alkenyl radical having up to 12 carbon atoms which is unsubstituted, substituted by one or two fluorine atoms, or at least one substituent by halogen, where one or more CH₃ groups in these radicals may also, in each case independently of one another, be replaced by a group defined by



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the EHP effect, and the LCD in plane switching displays.

The principle of electrically controlled switching in the EHP effect, or alternatively IAD effect, in liquid-crystal phases, was described for the first time in 1971 (K.K. Schreckel and K. Fahrenschon, "Deformation of nematic liquid crystals with vertical orientation in electrical fields", Appl. Phys. Lett. 18, 1971, 1011). This was followed by papers by K.K. Fahn, Appl. Phys. Lett. 20, 1972, 1011, and M. Iannace and J. Kober, Appl. Phys. 44, 1973, 4600.

The papers by J. Fabert and M. Clerc (SID 81 Digest Techn. Papers, 1981, 3), J. Duchene (Displays 1981, 3) and H. Schadt (SID 82 Digest Techn. Papers, 1982, 344) have shown that liquid crystalline phases must have high values for the ratio between the elastic constants K_2/K_1 , high values for the optical anisotropy Δn and values for the dielectric anisotropy $\Delta\epsilon$ of from $+10$ to $+4$ in order to be suitable for high information display elements based on the EHP effect. Electro-optical display elements based on the EHP effect have a nematic phase edge alignment. Conventionally negative liquid crystal media can also be used in displays utilizing the so-called IPS effect.

Technical use of this effect in electro-optical display elements requires 12 phases with most satisfactory reliability of operation. Particularly important here are electrical resistance, mechanical and physical stresses with an heat radiation in the illuminated window and thermal expansion and contraction of the material.

Technically suitable materials are therefore required to have a low electrical resistance, low thermal expansion, low contraction, low heat radiation and low thermal conductivity.

There are two classes of liquid crystal display: one which have been fabricated without individual addressing and which meet all these requirements. In general, these are prepared in large quantities preferably in rectangular shape and are prepared in order to obtain ribbons which can be used as display phases. However, optimum phases could not be prepared easily in this way, since the liquid crystalline materials are simultaneously sensitive to electric and magnetic fields.

Matrix liquid crystal displays are known. The linear elements which can be used for individual switching in the individual pixels are, for example, active elements (i.e. transistors). This is then referred to as an "active matrix", and a distinction can be made between two types:

1. Thin film wide semiconductor transistors on a silicon wafer as substrate.
2. Thin film transistors (TFT) on a glass plate or substrate.

In the case of type 1, the electro-optical effect used is usually dynamic scattering or the most heat effect. The use of angle crystal material as the substrate material limits the display area, since even smaller areas will lead to part display similar in problems to the active.

In the case of type 2, the electro-optical effect used is usually the TN effect.

A distinction is also between the active and TFT transistors and the TFT transistors. For example, the TFT transistors are prepared by the TFT process.

typical mixture generally keeps over the life of an MLC display without interaction with the interior surfaces of the display. A more limited resistance is very important for displays which must have acceptable resistance values over a long service life.

The disadvantages of the MLC-III displays described hitherto are due to their comparatively low contrast, relatively small viewing angle dependence and the difficulty of generating gray shades in these displays.

MLC-III and MLC-IV displays based on the WFB effect. However, the MLC mixtures described therein, which are based on α -alkylphenyl derivatives containing an ester, ether or ethyl bridge, have low "voltage holding ratio" (Hk) values after UV exposure.

There thus continues to be a great demand for MLC displays which have very high resistivity at the same time as a broad operating temperature range, short response times and a low threshold voltage which can be used to produce gray shades.

It is an object of the invention to provide MLC displays based on the WFB effect which do not have the disadvantages described above, or only to a lesser extent, and which have other very high advantages.

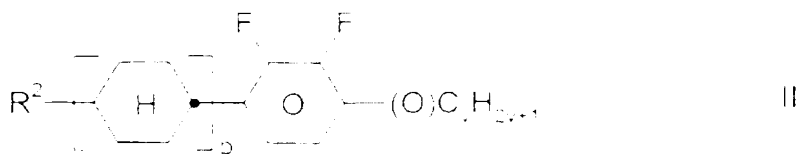
It has now been found that there is still a great demand for liquid crystal mixtures comprising at least one compound of the formula II and one compound of the formula III as described in the display elements.

The present invention relates to a liquid crystal mixture comprising at least one compound of the formula II and one compound of the formula III as described in the display elements, and to a display element comprising such a mixture.

The mixture according to the invention has very favorable values for the operative threshold, relatively high values for the killing rate and at the same time very good low temperature stability.

Some preferred embodiments are mentioned below:

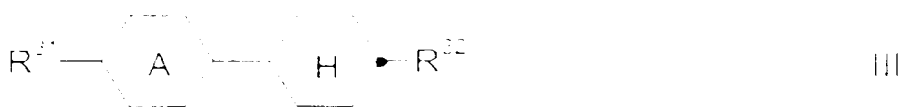
- a) A medium which additionally comprises one or more compounds of the formula II:



in which

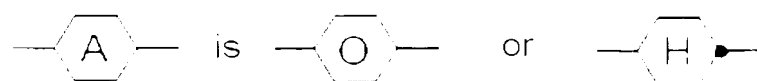
- R² is independently as defined for R¹, R³ and R⁴,
 n is 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98, 99, 100,
 is from 1 to 8.

- b) A medium which additionally comprises one or more compounds of the formula III:

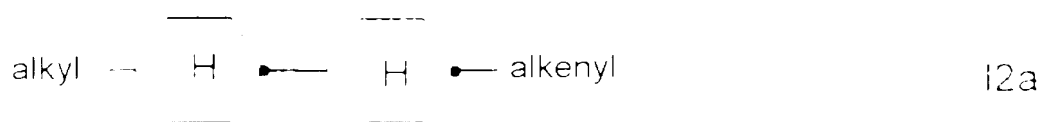


in which

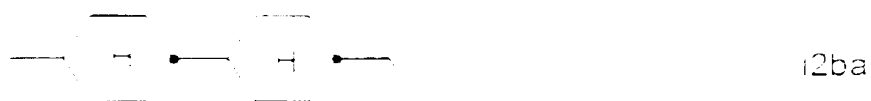
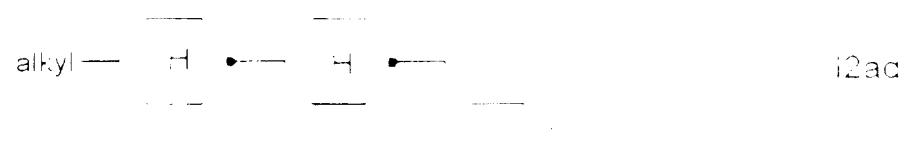
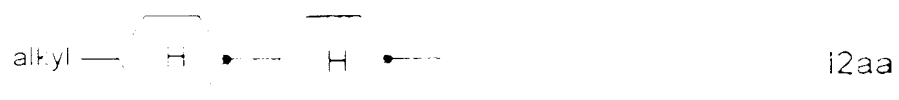
- R²¹ and R²² are independently as defined for R¹, R², R³, R⁴, R⁵, R⁶, R⁷, R⁸, R⁹, R¹⁰, R¹¹, R¹², R¹³, R¹⁴, R¹⁵, R¹⁶, R¹⁷, R¹⁸, R¹⁹, R²⁰, R²¹, R²², R²³, R²⁴, R²⁵, R²⁶, R²⁷, R²⁸, R²⁹, R³⁰, R³¹, R³², R³³, R³⁴, R³⁵, R³⁶, R³⁷, R³⁸, R³⁹, R⁴⁰, R⁴¹, R⁴², R⁴³, R⁴⁴, R⁴⁵, R⁴⁶, R⁴⁷, R⁴⁸, R⁴⁹, R⁵⁰, R⁵¹, R⁵², R⁵³, R⁵⁴, R⁵⁵, R⁵⁶, R⁵⁷, R⁵⁸, R⁵⁹, R⁶⁰, R⁶¹, R⁶², R⁶³, R⁶⁴, R⁶⁵, R⁶⁶, R⁶⁷, R⁶⁸, R⁶⁹, R⁷⁰, R⁷¹, R⁷², R⁷³, R⁷⁴, R⁷⁵, R⁷⁶, R⁷⁷, R⁷⁸, R⁷⁹, R⁸⁰, R⁸¹, R⁸², R⁸³, R⁸⁴, R⁸⁵, R⁸⁶, R⁸⁷, R⁸⁸, R⁸⁹, R⁹⁰, R⁹¹, R⁹², R⁹³, R⁹⁴, R⁹⁵, R⁹⁶, R⁹⁷, R⁹⁸, R⁹⁹, R¹⁰⁰.



- e. A medium which comprises two, three, four, or more, preferably two, three or four, compounds of the formula II.
- f. A medium which comprises at least two compounds of the formula II.
- g. A medium in which the proportion of compounds of the formula II in the total mixture is at least 1% by weight, preferably at least 2% by weight.
- h. A medium in which the proportion of compounds of the formula II in the total mixture is at least 5% by weight, preferably at least 10% by weight.
- i. A medium in which the proportion of compounds of the formula II in the total mixture is at least 10% by weight.
- j. A medium in which the proportion of compounds of the formula III in the total mixture is at least 1% by weight.
- k. A medium in which the proportion of compounds of the formula III in the total mixture is at least 5% by weight.
- l. A medium which comprises at least one compound selected from the formula I, II and III.



particular preference is given to the ring ends of the molecules (12aa-12af and 12ba-12be):

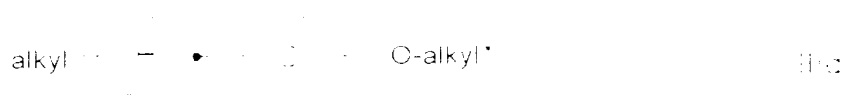
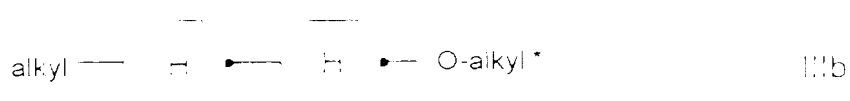
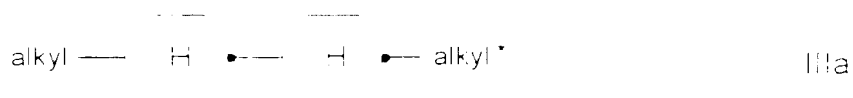


alkenyl and

alkenyl* are each, independently of one another,
a straight-chain alkenyl radical having
1-6 carbon atoms, and

alkyl is a straight-chain alkyl radical having
1-6 carbon atoms.

A medium which additionally comprises a compound
selected from the formulae IIIa to IIId:



in which

alkyl and

alkyl* are each, independently of one another,
a straight-chain alkyl radical having 1-6 carbon
atoms.

The medium according to the invention preferably
comprises at least one compound of the formula
IIIa and of formula IIIb.

It is also possible to employ the medium

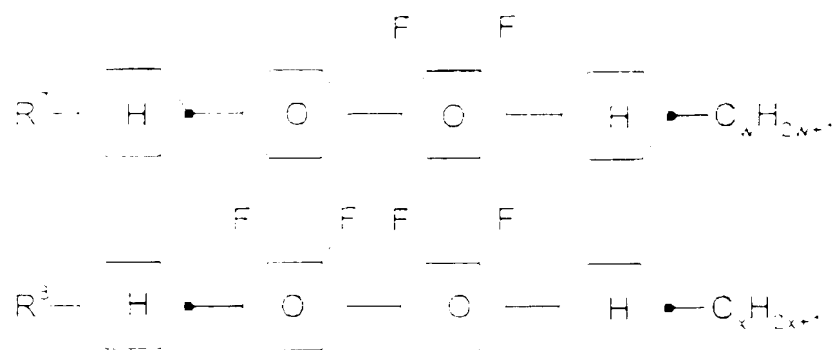
in which the alkyl and alkyl* radicals are
phenyl radicals.

by weight of the various components of the formula II.

and

by weight of the various components of the formula III.

- (c) A mixture which substantially comprises one more component of the formula

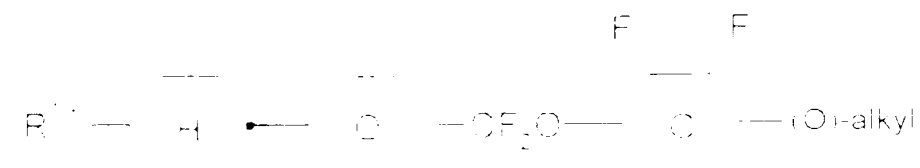
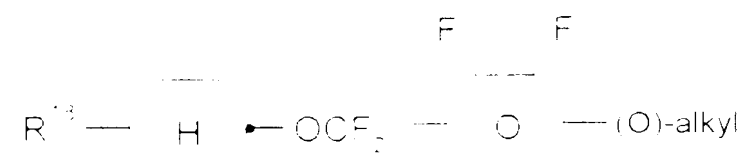
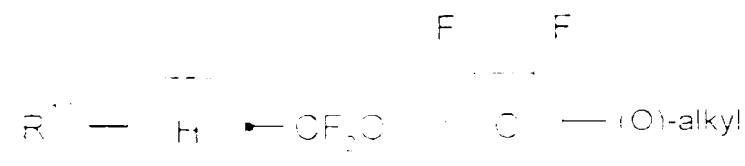
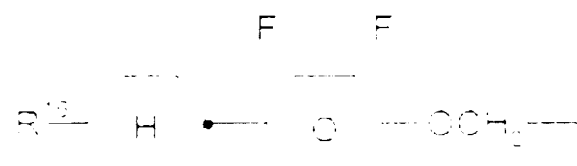
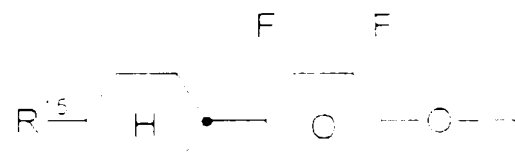
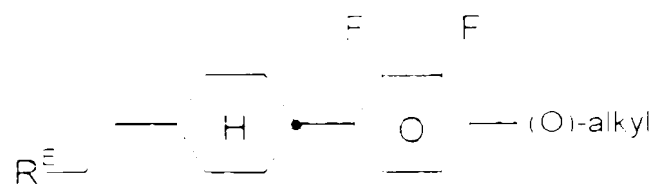
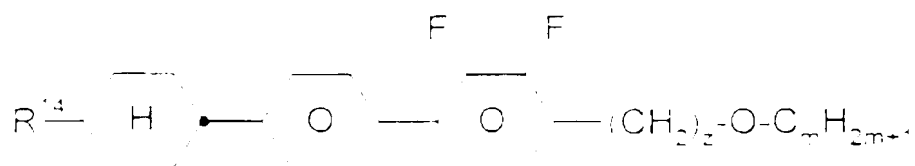
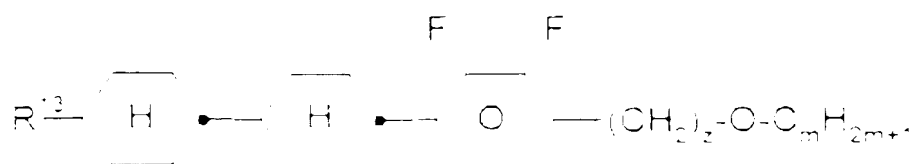


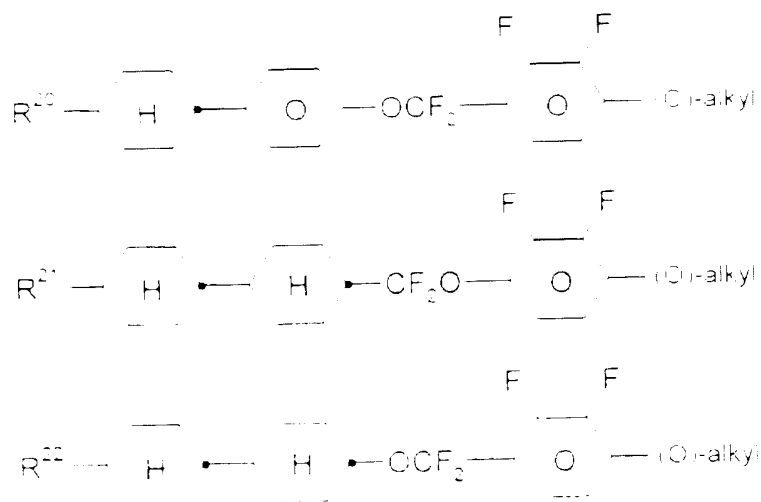
in which

F and F' are each, independently of one another, an alkyl or an aryl group and R and R' are each an alkyl group.

x and x' are each, independently of one another, an integer from 1 to 10.

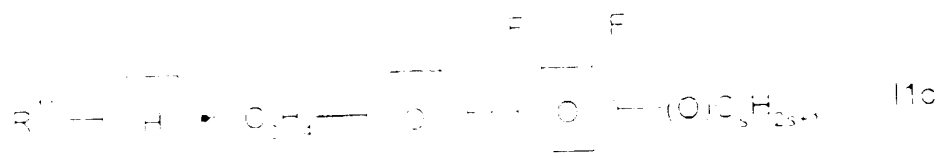
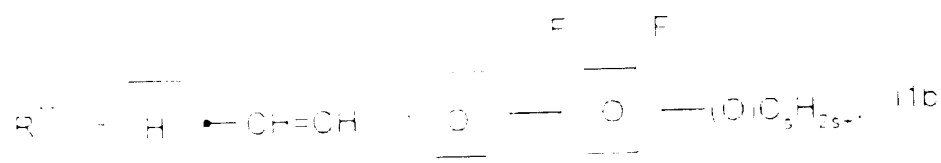
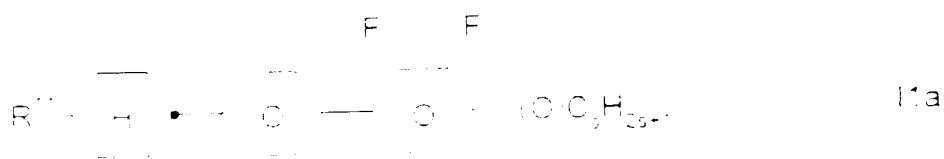
- (d) A mixture which substantially comprises one more component of the formula

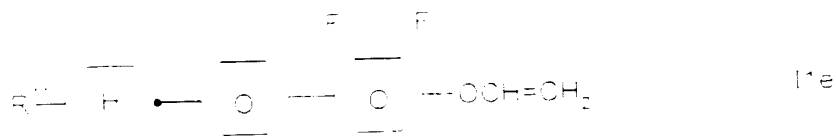




ii. which R^{20} and R^{21} are each, independently of one another, as defined for R^1 , R^2 and R^3 , and n and m are each, independently of one another, 1-6, R^{22} is H , CH_3 , CH or $n\text{-C}_4\text{H}_9$.

iii. A medium in which the compound of the formula II is selected from the group consisting of IIa to IIc:





based on standard methods which are described in the literature.

The present liquid crystal mixtures in the displays according to the invention generally comprise two components A and B, which themselves consist of one or more individual compounds.

Component A has significantly negative dielectric anisotropy and gives the nematic phase a dielectric anisotropy of ≤ -1.5 . It preferably comprises compounds of the formulae II and III.

The proportion of component A is preferably between 45 and 100%, in particular between 60 and 100%.

For component A, one or more individual compounds having a $\Delta\epsilon \leq -1.5$ are preferably selected. The smaller the proportion of component A in the total mixture, the more negative this value must be.

Component B has pronounced nematogenicity and a flow viscosity of not more than 2 mm s⁻¹, preferably not more than 0.5 mm s⁻¹, at 25°C. It preferably comprises compounds of the formulae II and III.

Particularly preferred individual compounds of component B are extremely low-viscosity nematic liquid crystals having a flow viscosity of not more than 15 mm s⁻¹, preferably not more than 1.5 mm s⁻¹, at 25°C.

Component B has monotropic or enantiotropic nematogenicity, has nematic phases and can prevent the appearance of smectic phases in liquid crystal mixtures from a very low temperature. If, for example, a monotropic liquid crystal mixture is mixed with an enantiotropic liquid crystal mixture, the latter is suppressed at one temperature that is shifted to a lower temperature compared to the onset of the nematic phase.

Nematic liquid-crystalline materials are known in the prior art skilled in the art from the literature. Particular preference is given to compounds of the formula III.

In addition, these liquid crystal phases can also contain more than 10 components, preferably from 15 to 20 components.

The phases preferably contain from 4 to 18, in particular 5 to 12, compounds of the formulae II, III, IV and optionally V.

Besides compounds of the formulae II, III, IV and V, it is also possible for other constituents to be present, for example in an amount of up to 40% of the total mixture, but preferably up to 10%, in particular up to 5%.

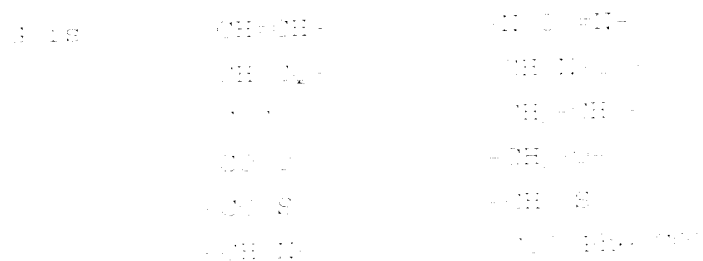
The other constituents are preferably selected from nematic or nematicogenic substances, in particular known substances from the classes consisting of the anoxy-benzenes, benzylidenemalines, biphenyls, terphenyls, phenyl or cyclohexyl benzenes, phenyl or cyclohexyl cyclohexanecarboxylates, phenylcyclohexanes, cyclohexylbiphenyls, cyclohexylcyclohexanes, cyclohexylnaphthalenes, 1,4-bis-cyclohexylbiphenyls or cyclohexylpyrimidines, phenyl or cyclohexylidene, optionally halogenated stilbenes, acyl phenyl ethers, alanes and substituted succinic acids.

Important compounds which can be used as constituents of liquid crystal mixtures of this type can be found, for example, in Table IV.

Table IV

Compounds which are known as liquid-crystalline materials are listed in Table IV. The compounds are listed in the order of increasing molecular weight. The compounds are listed in the order of increasing molecular weight.

disubstituted biphenyl, phenylcyclohexane and cyclohexylcyclohexane systems, 1,1-disubstituted pyridine and 1,1-dioxane rings, 1,1-disubstituted naphthalene, 1,1- and tetraaryl naphthalene, pinacoline and tetraaryl pinacoline.



or a C-C single bond. Q is halogen, preferably chlorine, or CN, and R and R' are each alkyl, alkenyl, alkoxy, alkanoyloxy or alkoxycarbonyloxy having up to 18, preferably up to 6, carbon atoms, or one of these radicals is alternatively CN, NO, NO₂, NCS, CF₃, F, Cl or Br.

In most of these compounds, R and R' are different from one another. One of these radicals usually bears an alkyl or alkoxy group. However, the variants of the proposed substituents are also common. Many such substances or mixtures thereof are commercially available. All these substances can be prepared by methods known in the art from the literature.

It will be appreciated by a person skilled in the art that the R/R' mixture according to the invention may also comprise compounds in which, for example, R, R' or R and R' have been replaced by the corresponding isotopes.

The invention also relates to liquid crystal mixtures consisting of the compounds according to the invention and at least one other compound and a solvent or diluent. For example, the mixture may contain:

With a number of variations, it is believed that one skilled in the art can, using the preceding description, outline the present invention to the fullest extent. The following preferred specific embodiments are, therefore, to be construed as merely illustrative, and not limitative of the remainder of the disclosure in any way whatsoever.

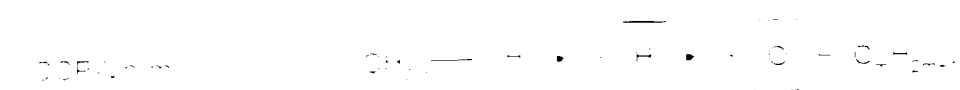
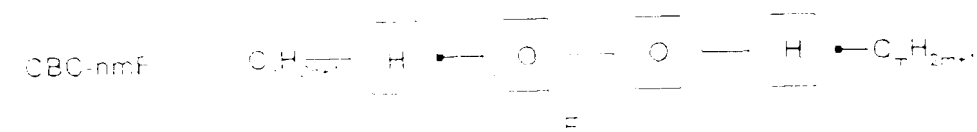
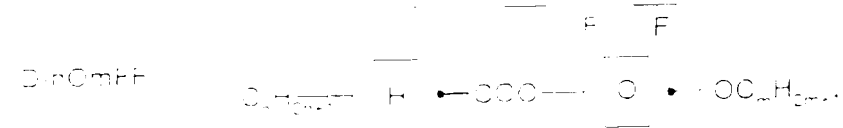
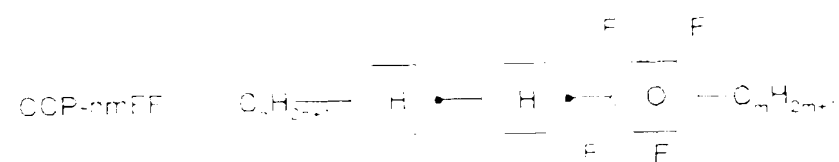
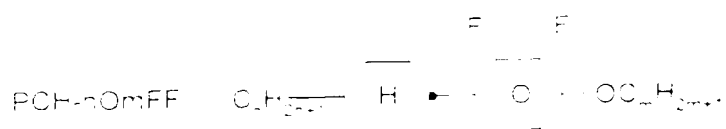
In the foregoing and in the following examples, all temperatures are set forth uncorrected in degrees Celsius; and, unless otherwise indicated, all parts and percentages are by weight.

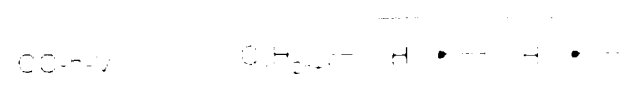
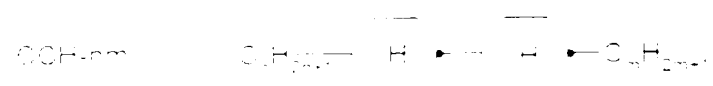
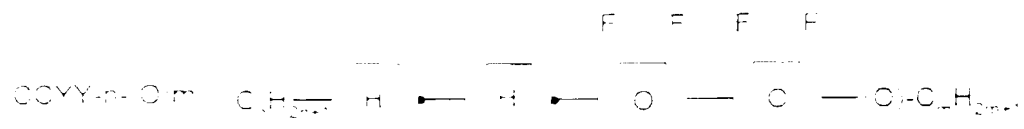
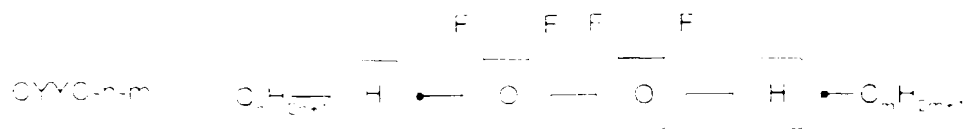
The entire disclosure of all applications, patents and publications, cited above, and of corresponding German application No. DE 11, 15, 493.1, filed April 14, 1971, is hereby incorporated by reference.

Besides the compounds of the formulae 11 and 12, the liquid-crystal mixtures according to the invention preferably comprise one or more of the compounds mentioned below.

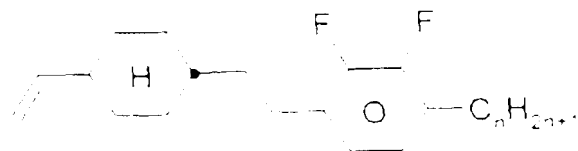
The following abbreviations are used:

13 m = 1-6; n = 1-6

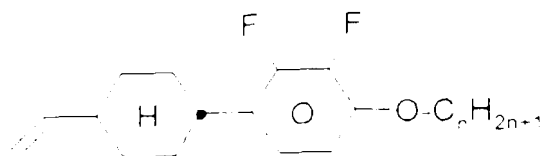




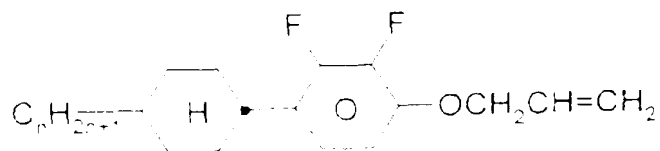
CVY-V-n



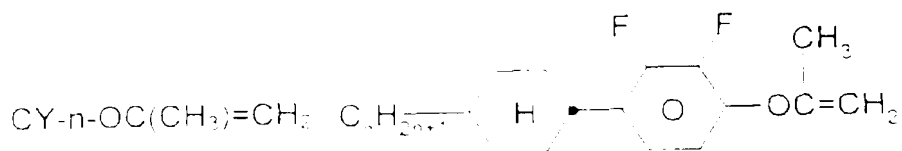
CY-V-On



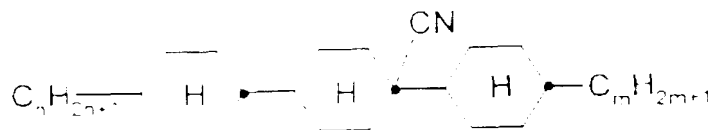
CY-n-O1V



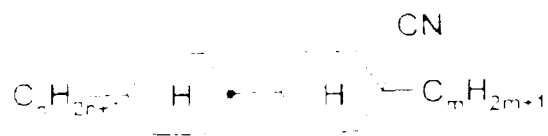
CY-n-OC(CH3)=CH2



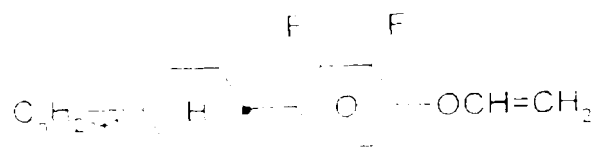
BCN-nm



CCN-nm



CY-n-OV



PCH-nm



PCH-nOm



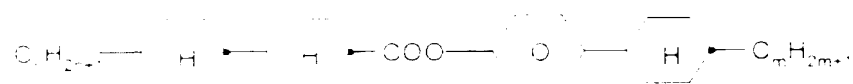
PGIGI-n-F



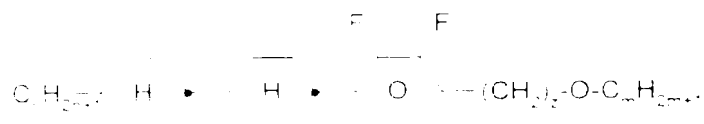
BCH-nm



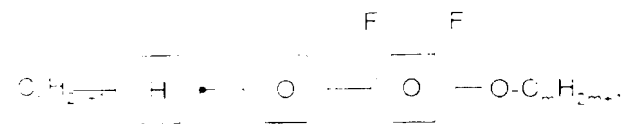
CCPC-nm



CCY-n-zOm



CPY-n-Om



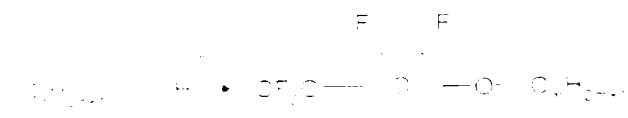
CPY-V-Om



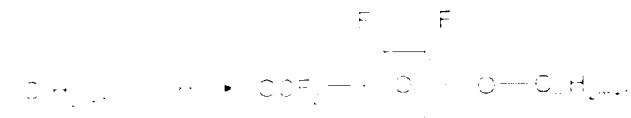
CPY-nm



CCY-n-Om



CCY-n-Om





Next day examples

Example 1

10H + 4PF	10.1	8 + 11	8 + 4 = 12
10H + 4PF	10.1	Clearing up and 10.1	10.1
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	8 + 11 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	7 + 10 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	10.1	10.1
10H + 4PF	10.1	10.1 in cells: 10.1 mm, 10.1	10.1 11
		At: -10° C, -3° C, -4° C	

Example 2

10H + 4PF	10.1	8 + 11	8 + 4 = 12
10H + 4PF	10.1	Clearing up and 10.1	10.1
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	8 + 11 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	7 + 10 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	10.1	10.1
10H + 4PF	10.1	10.1 in cells: 10.1 mm, 10.1	10.1 11
10H + 4PF	10.1	At: 10.1 and 10.1	10.1 11

Example 3

10H + 4PF	10.1	8 + 11	8 + 4 = 12
10H + 4PF	10.1	Clearing up and 10.1	10.1
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	Am. (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	8 + 11 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	7 + 10 (10.1 mm, 10.1) 10.1	10.1 11
10H + 4PF	10.1	10.1	10.1
10H + 4PF	10.1	10.1	10.1
10H + 4PF	10.1	10.1	10.1
10H + 4PF	10.1	10.1	10.1

[illegible]

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Example 10

$10H + 4PF$	14.	$Z = 11$	4.0×10^{-10}
$10H + 1PF$	14.	Clearing point, 10^4 G	7.0
$10H + 4PF$	14.	An. (1.5 nm, 10^4 G)	4.0×10^{-10}
$10H + 1PF$	14.	Ac. (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	ϵ (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	γ (1000 G, 10^4 G)	1.0
$10H + 1PF$	14.	η (1000 G)	1.0
$10H + 1PF$	14.		
$10H + 1PF$	14.		
$10H + 1PF$	14.		

Example 10.1

$10H + 4PF$	14.	$Z = 11$	4.0×10^{-10}
$10H + 1PF$	14.	Clearing point, 10^4 G	7.0
$10H + 4PF$	14.	An. (1.5 nm, 10^4 G)	4.0×10^{-10}
$10H + 1PF$	14.	Ac. (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	ϵ (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	γ (1000 G, 10^4 G)	1.0
$10H + 1PF$	14.	η (1000 G)	1.0
$10H + 1PF$	14.		
$10H + 1PF$	14.		

Example 10.2

$10H + 4PF$	14.	$Z = 11$	4.0×10^{-10}
$10H + 1PF$	14.	Clearing point, 10^4 G	7.0
$10H + 1PF$	14.	An. (1.5 nm, 10^4 G)	4.0×10^{-10}
$10H + 1PF$	14.	Ac. (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	ϵ (1 kHn, 10^4 G)	3.0
$10H + 1PF$	14.	γ (1000 G, 10^4 G)	1.0
$10H + 1PF$	14.	η (1000 G)	1.0
$10H + 1PF$	14.		
$10H + 1PF$	14.		
$10H + 1PF$	14.		

[illegible]

Figure 1 is a schematic representation of the experimental design. It shows a sequence of events: a subject is presented with a stimulus (a face), then a response is recorded (a button press), and finally, the subject is presented with a feedback stimulus (a face). The sequence is labeled with 'Stimulus', 'Response', and 'Feedback'.

[illegible]

Example 10

PCH-1-4FF	10.10	Clearing point (°C):	70.6
PCH-1-4FF	10.10	Δn (10 ⁻⁶ mm ⁻¹ , 21°C):	0.00004
PCH-1-4FF	10.10	$\Delta \rho$ (1 kH ₂ , 21°C):	0.4
CPY-1-1	10.10	ϵ (1 kH ₂ , 21°C):	3.6
CPQT-1-1	5.10	η (mPa·s):	1.10
CPQT-1-1	5.10	γ (mPa·s, 21°C):	116
CPY-1-1	5.10		
BCH-10	5.10		
CC-1-1	5.10		
CC-1-1	5.10		
CC-1-1	10.10		

Example 11

PCH-1-4FF	10.10	Clearing point (°C):	70.6
PCH-1-4FF	10.10	Δn (10 ⁻⁶ mm ⁻¹ , 21°C):	0.00004
PCH-1-4FF	10.10	$\Delta \rho$ (1 kH ₂ , 21°C):	0.4
CPY-1-1	10.10	ϵ (1 kH ₂ , 21°C):	3.6
CPQT-1-1	5.10	η (mPa·s):	1.10
CPQT-1-1	5.10	γ (mPa·s, 21°C):	116
CPY-1-1	5.10		
BCH-10	5.10		
CC-1-1	5.10		
CC-1-1	5.10		
CC-1-1	10.10		

Example 12

PCH-1-4FF	10.10	Clearing point (°C):	70.6
PCH-1-4FF	10.10	Δn (10 ⁻⁶ mm ⁻¹ , 21°C):	0.00004
PCH-1-4FF	10.10	$\Delta \rho$ (1 kH ₂ , 21°C):	0.4
CPY-1-1	10.10	ϵ (1 kH ₂ , 21°C):	3.6
CPQT-1-1	5.10	η (mPa·s):	1.10
CPQT-1-1	5.10	γ (mPa·s, 21°C):	116
CPY-1-1	5.10		
BCH-10	5.10		
CC-1-1	5.10		
CC-1-1	5.10		
CC-1-1	10.10		

Example 17

PCH-514FF	11.1	Clearing point (°C):	6.1
PCH-512FF	11.1	Δn (589 nm, 20 °C):	+1.0311
PCH-514FF	11.1	Δn (11 KHz, 20 °C):	+1.6
CCQY-3-11	11.1	ϵ (11 KHz, 20 °C):	3.6
CPY-2-11	11.1	γ (°C):	2.16
CPY-3-11	11.1	γ (MPa·s, 20 °C):	11
CCQY-3	11.1		
CC-3-11	11.1		
CCN-3	11.1		
CC-3-11	11.1		

Example 18

PCH-514FF	11.1	Clearing point (°C):	7.5
PCH-512FF	11.1	Δn (589 nm, 20 °C):	+1.0311
PCH-514FF	11.1	Δn (11 KHz, 20 °C):	+3.7
CCQY-3-11	11.1	ϵ (11 KHz, 20 °C):	3.6
CPY-2-11	11.1	γ (°C):	2.16
CPY-3-11	11.1	γ (MPa·s, 20 °C):	11
CPY-11-1	11.1		
CC-3-11	11.1		
CCN-3	11.1		
CC-3-11	11.1		